Small-Angle X-ray Scattering at Large Angles in the Soft X-ray Range: Layer Discontinuity in Ultra-Short Period W/B_4C Multilayers

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INTRODUCTION

Recent novel applications of multilayer x-ray mirrors, for example as linear polarizers in the soft x-ray,[1] have brought with them new interest in multilayers of ultra-short periods (d < 15Å). Most materials combinations show a severe loss of reflectivity at periods below 30-40Å, but W/B_4C has been shown[2,3] to maintain useful reflectivities (1-2% or better) for periods down to 12Å. To optimize performance at these periods, we have studied the microstructure of W/B_4C multilayers with Small-Angle X-ray Scattering (SAXS) experiments at Beamline 6.3.2 at ALS. Measurements usually performed at 0.1°-5°in the hard x-ray range correspond to angles up to 60° at soft x-ray energies (700eV). While not generally applied to x-ray multilayers, this technique provides valuable information on in-plane inhomogeneities not easily accessible to other techniques. Discontinuity of the films at early stages of growth contributes to reflectivity loss and can be directly measured by this technique.

EXPERIMENT

The multilayers in this study were grown by standard magnetron sputtering techniques described elsewhere, on free-standing Si_3N_4 membranes about 1500Å thick. There were examined in transmission at BL 6.3.2 at E = 700eV and on a rotating-anode source at CuK_α (λ = 1.54Å) The scattered signal was measured by a detector at angle 2θ, and the sample was rotated by θ to keep the scattering vector in the plane of the films. Background signal from the tails of the incident beam was measured separately and subtracted, and the remaining signal divided by the full beam intensity I_0 to yield an absolute scattered intensity vs. angle. Multilayers of period d = 6, 14, 22 and 30Å were measured.

ANALYSIS

The scattered signal was compared to a simple SAXS model based on earlier work by Rice.[4] The model assumes spherical particles of one composition in a homogeneous matrix of another. Using the known overall multilayer composition, the size of the particles and the volume fraction they occupy were varied to fit the experimental results. Within plausible ranges of these parameters a unique fit was obtained.

RESULTS AND DISCUSSION

The experimental data (points) and fits (smooth curves) are shown in Fig. 1. For the shortest d-spacing, a broad peak is present at both incident energies. At d = 14Å a weak peak at a smaller angle (corresponding to larger particles) is present, and at d = 22Å and d = 30Å no SAXS signal is observed. This is consistent with a transition from discontinuous to continuous multilayers between d = 14Å and D = 22Å. Because the scattering factor for W is more than ten times that of B or C at both wavelengths, the SAXS signal represents almost exclusively the W-W correlations. For a continuous multilayer the scattering vector lies parallel to the layers and measures only weak in-plane density fluctuations, while for a discontinuous multilayer the sizes and separations of the islands in the film give a strong in-plane signal.

The fit parameters agree well for the two incident energies used – particles of about 25Å diameter for the d = 6Å multilayer and 32Å diameter for the d = 14Å multilayer. The results are corroborated by plan-view HRTEM of other W/B_4C specimens grown under the same conditions – Fig. 2. shows a d = 6Å multilayer with a clear islanded structure. The islands are of irregular shape, consistent with a broad distribution of widths indicated by the peak width in the SAXS signal, and show diameters about 25Å.

CONCLUSIONS

W/B_4C forms layered structures with separate phases for periods as short as 6Å, though for periods below 14-22Å the layers are discontinuous. This work demonstrates that scattering measurements in the soft x-ray can probe inhomogeneities in materials on the nm scale and greater. Coupled with the strong anomalous dispersion (in charge and possibly magnetic scattering) associated with many soft x-ray core levels, this capability can be readily extended to study a variety of materials and problems.

REFERENCES

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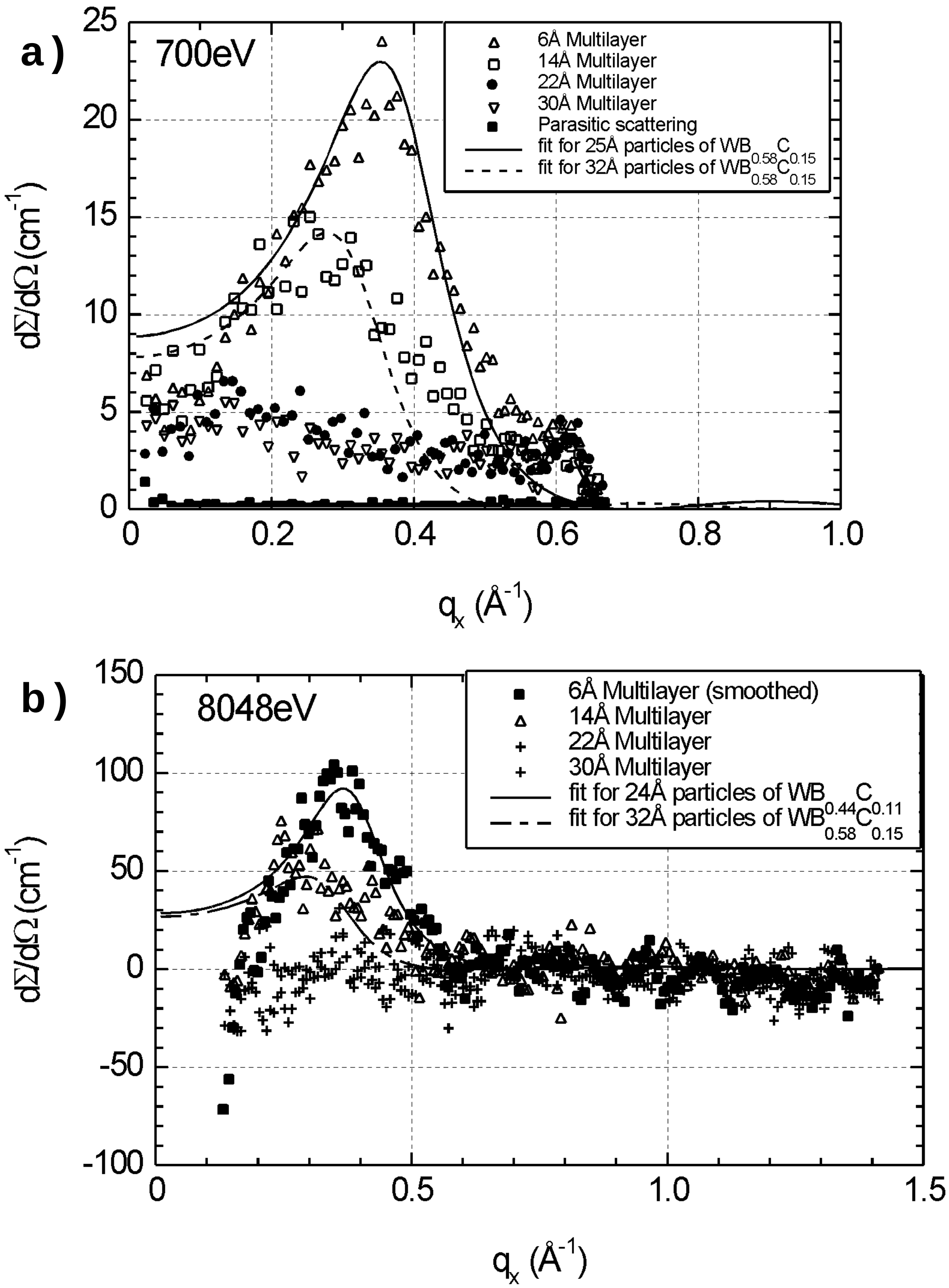

Fig. 1. *a)* Small-Angle X-Ray Scattering (SAXS) data (points) and fits (smooth curves) for W/B_4C multilayers of four periods, taken at E = 700eV. Fits are consistent with the W-rich layers in the shortest-period multilayers (6Å and 14Å) consisting of islands of about 24Å and 32Å diameter rather than continuous layers. The islands are not bulk W but compounds near the composition W_2B. *b)* Similar results taken at E = 8048eV (CuK_{α}).

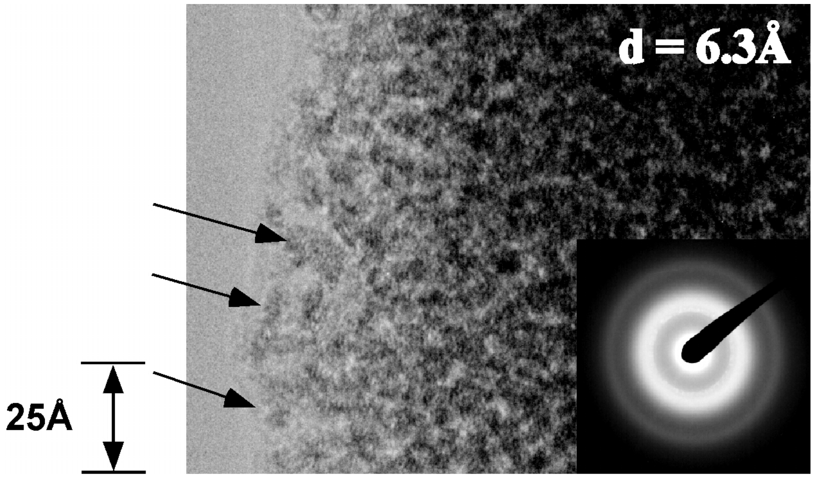

Fig. 2. Plan-view HRTEM of d = 6Å W/B_4C multilayer. Island structure of W-rich layers is clearly visible.

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